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TITLE: STATUS OF ^{241}AM RECOVERY AND PURIFICATION AT LOS ALAMOS SCIENTIFIC LABORATORY

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NOTICE

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STATUS OF ^{241}Am RECOVERY AND PURIFICATION AT THE

LOS ALAMOS SCIENTIFIC LABORATORY

By

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ABSTRACT

Americium recovery was initiated at Los Alamos
Scientific Laboratory (LASL) in the late 1940's. The
early procedures separated gram quantities of americium
from large amounts of impurities including plutonium and
the rare earths. Ion exchange procedures were developed
for further purification.

Until recently, no routine processing of americium
has been done at LASL for several years. The increasing
demand for americium in oil-well logging instruments and
other uses led LASL to develop and install a process to
recover larger quantities of americium. The LASL pro-
cess was developed around the chemistry of americium
that had been elucidated both at LASL and at other fa-
cilities.

Presently, the americium feed is obtained as a by-
product from a plutonium purification process at the new
plutonium facility at LASL. This feed filtrate from a
peroxide precipitation process is precipitated as a
slurry of hydroxides, filtered, dissolved in nitric
acid, and passed through an anion exchange column to
remove any residual plutonium. The americium, con-
tained in the effluent, is precipitated as the oxalate
and calcined to the oxide.

Americium is also available in other highly salted
acidic process streams. These should lend themselves
to solvent extraction. Developmental work has been
promising, and a dibutyl butyl phosphonate-kerosene ex-
traction process is being brought on-line.

HISTORY

Separation of americium from parent plutonium began at LASL in late 1947 with the formation of a small group for that purpose headed by R. A. Penneman. The early investigations centered necessarily on the isolation of americium from plutonium which had sufficient irradiation and age that the beta decay of 14 year ^{241}Pu would yield appreciable ^{241}Am (150 - 200 mgs/Kg of Pu). Some kilograms of plutonium turnings were processed by us yielding about a gram of americium. That plutonium had been through the $BiPO_4/LaF_3$ process and contained a hundred times as much lanthanum as americium; this lanthanum naturally separated with the crude americium. Later, filtrates were processed from the plutonium peroxide process used in the production plant. Plutonium that had been through Purex gave cleaner americium.

Americium was isolated first from plutonium, then from lanthanum and other impurities, by a combination of precipitation, solvent extraction, and ion exchange processes. Parallel with the separation, a vigorous program of research began. Beginning in 1950, a series of publications[1-24] on americium put into the world literature much of the classic chemistry of americium: the hexavalent state, the soluble tetravalent state, oxidation potentials, disproportionation, the crystal structure(s) of the metal, and many compounds of americium. In particular, our use of peroxydisulfate or ozone to oxidize americium to the (V) or (VI) states still provides a basis for americium removal from other elements by certain techniques. Irradiation of americium, first at Chalk River and later at the MTR (Idaho), gave us curium for study. Indeed, the oxidation of americium and its separation from curium provided the clue utilized by others in a patented process for separation of americium from the rare earths.

PRODUCTION

At present, americium is separated and purified in larger quantities at the new plutonium facility (TA-55) at LASL (Figure 1). The feed for the americium production comes from a plutonium oxide production line. This line produces high purity ceramic grade plutonium oxide for the Fast Flux Test Facility (FFTF) at Richland, Washington. The feed for this plutonium oxide process is aged plutonium metal and has a sizable amount of americium which has grown in during storage.

The flowsheet for the FFTF plutonium oxide production is shown in Figure 2. Briefly, the plutonium metal is converted to an impure oxide by

burning the metal in air. This is followed by dissolution of the impure oxide in a concentrated nitric acid-hydrogen fluoride solution. The americium is separated from the plutonium by precipitation of the plutonium as the peroxide. Americium does not form an insoluble peroxide and stays in the filtrate with other cationic impurities. The active peroxide filtrate is slowly dripped into 9 $\underline{M}$ NaOH. The combination of strong caustic and heat destroys the peroxides and precipitates the americium as the hydroxide. Any residual plutonium in the filtrate, along with other cations, is precipitated also as the hydroxide. The flowsheet for the americium oxide production is shown in Figure 3.

The cooled slurry of hydroxides is transferred by vacuum into glass tanks (Figure 4). These tanks are shielded with Teflon coated lead to minimize the radiation exposure to the technicians. The lead is Teflon coated to prevent contamination with lead in the americium product. The slurry of hydroxides is gelatinous in nature and is difficult to filter. To improve the filterability of these hydroxides, the slurry is allowed to stand overnight and settle (Figure 5). The hydroxides seem to aggregate upon standing. The filtering process is also faster as the clear supernant liquid above the aggregated hydroxides can be passed through the filter very fast. The remaining hydroxides are then slowly filtered with vacuum onto filter paper in a 20 cm stainless steel filter boat (Figure 6), rinsed with 0.1 $\underline{M}$ NaOH to remove excess sodium salts, and redissolved in concentrated nitric acid. After analysis for Am and Pu, the filtrate from this filtration is sent to caustic waste. The filter paper is rinsed well with water, dried, and incinerated. The composition of two representative batch-are shown in Table 1.

The next step in production is removal of residual plutonium from the americium by ion exchange chromatography. The americium nitrate solution is passed, after acidity adjustment to 7.5 $\underline{M}$, through a 6" x 12" ion exchange column containing Dowex 1 (anion resin). Periodically, an unknown gel (thought to be a silica matrix with occluded sodium nitrate) is observed in the americium nitrate and must be filtered off to prevent plugging of the ion exchange column. The ion exchange column is gravity fed. It is also shielded with a layer of Teflon coated lead to reduce radiation exposure of workers. The usual problems of running concentrated americium solutions through ion exchange resins are encountered. An example is the gas generation from radiolysis of the solution. The ion exchange column

is run on a batch schedule. Concentrated americium solution is never allowed to stand on the column. After a batch has passed through the column, the americium nitrate solution remaining in the column is replaced with enough 7 $\underline{M}$ nitric acid solution to wash out all of the americium. This usually requires two void volumes of 7 $\underline{M}$ HNO_3. After several batches of americium have been passed through the ion exchange column, the anion resin becomes loaded with $Pu(NO_3)_6^=$ and must be eluted. The acidity of the solution on the ion exchange column is lowered to approximately 1 $\underline{M}$, and the plutonium is eluted with a hydroxylamine nitrate solution. It is worthy to note the violent reaction that occurs between hydroxylamine nitrate and concentrated nitric acid! Therefore, it is imperative that the acidity on the column be reduced before starting to elute. The eluted plutonium solution is sent elsewhere in the plutonium facility for further processing. The anion resin is frequently replaced with new resin to prevent buildup of resin degradation products.

Before the americium can be precipitated as the oxalate, the acidity of the solution must be lowered. This cannot be done by the addition of NaOH or KOH as these cations are carried down with the americium oxalate. The acidity adjustment can be made with NH_4OH with no product contamination, but processing problems resulting from ammonium vapors mixing with nitric acid fumes should be avoided. Even with the use of efficient traps, some ammonium vapors escape to form ammonium nitrate which plugs glovebox exhaust filters; plus, ammonium nitrate may sublime and condense past the filter throughout the entire exhaust system or may form by gaseous reaction after it has passed the filter.

The above mentioned problems make it highly desirable to lower the acidity of the ion exchange effluent with only distilled water. Volume constraints of equipment make it impractical to dilute seven molar of HNO_3 down to half molar solution. Some method of denitrification must be used to remove some of the HNO_3 before the final adjustment is done with water. This denitrification is accomplished by simple distillation (Figure 7). Approximately a ten fold reduction, both in volume and total moles of HNO_3, is achieved by distilling five liters of the ion exchange effluent to 0.5 liters. Before the reduced volume of americium nitrate completely cools to ambient temperature and salts out, enough distilled water is added to keep everything in solution. During this volume reduction, the americium has also been concentrated and appropriate shielding must be used.

The concentrated americium nitrate solution, now approximately 2 $\underline{M}$ in HNO_3, is transferred by vacuum into a stainless steel 2 liter bottle and transported to the oxalate precipitation process. The americium oxalate precipitation vessel is shown in Figure 8. The americium nitrate solution is transferred into the precipitation vessel by vacuum. The desired acidity is attained (0.5 $\underline{M}$) by the addition of distilled water. The americium is then precipitated with an excess of oxalic acid. At present, no in-line instrument is available for determining the exact concentration of americium in each batch; therefore, an excess of oxalic acid must be used in insure complete precipitation. These conditions allow some of the americium to be lost as a soluble oxalato complex whenever excess oxalic acid is available.

The americium oxalate is allowed to digest, with stirring, for at least one hour to minimize post precipitation. After digestion, the americium oxalate is filte.:d onto a Kynar frit in a stainless steel filter boat (Figure 9). The precipitate is washed with 0.1 $\underline{M}$ oxalic acid and dried by pulling air through the frit.

Calcination of the americium oxalate to the oxide is done in two stages. The americium oxalate is first heated in a quar'.z beaker on a flask heater, gently at first to prevent material from being carried out of the beaker by the decomposition products of oxalate and then more strongly until the yellow oxalate has entirely blackened. A final calcination of the mixture is done in a platinum dish inside a muffle furnace at 800°C for four hours to insure complete conversion to americium oxide. If necessary, the americium oxide is quickly ground to a fine powder in a mortar and pestle and resubmitted for calcination in the muffle furnace.

Finally, the black-brown americium oxide ir sieved through a 200 mesh 3 inch screen on a Fritsch analysette mechanical shaker. A complete analysis is done to determine whether the AmO_2 meets the required product specifications. The analysis of representative batches of americium oxide produced at LASL is shown in Table 2. Radiochemistry, emission spectroscopy, and spark source mass spectrometry[26] are used in the analysis of the AmO_2. The AmO_2 that meets all of the product specifications is prepared for shipment. Product not meeting these specifications is recycled.

Americium oxide prepared at LASL is shipped in the configuration shown in Figure 10. The AmO_2, in 25 gram quantities, is placed inside the stainless steel container and decontaminated. It is then placed into a plastic bag which is sealed with tape and decontaminated. This is bagged out of the

glovebox, placed in an open hood, plastic bag removed, and the stainless
steel container decontaminated once again. This is sealed in a plastic
bag, wrapped in steel wool, and canned in a lead lined food pack can.
This can is canned in a second food pack can. This configuration is
stored in the vault until shipment.

FUTURE AMERICIUM PRODUCTION

I. New Americium Source Stream

At LASL, the effluent from the production Pu ion exchange columns is
about 7 $\underline{M}$ HNO_3 and contains salts resulting from up-stream processing Am
and small amounts of Pu. This stream feeds the evaporators that recycle
HNO_3 and produce bottoms that are concentrated in salts that include the Am.
As these bottoms are cooled, some of the salts crystallize and are readily
separated from the supernatant liquid. This supernate, ∿ 7 - 9 $\underline{N}$ in HNO_3,
is made basic by NaOH addition causing most of the metals to precipitate as
the hydroxides. Filtration then gives a filtrate that can be discarded plus
a hydroxide cake. Presently, the hydroxide cake and the crystallized salts
from the bottoms are put into 20 year retrievable storage.

It has been determined that the bulk of any Am in the original evapora-
tor feed ends up in the supernate, hence in the hydroxide cake. This is con-
sidered as a potential Am source for the AmO_2 production line.

Examination of the data from the analyses of the supernate and hydroxide
cake shows them to contain primarily Na, Al, Mg, Ca, Fe, with some Pb, U,
and Pu plus the Am. Therefore, recovery of the Am from either the supernate
or redissolved hydroxide cake involves its separation from a highly salted
solution of these cations.

These conditions are valuable in solvent extraction techniques. It is
known[26-30] that Am extraction with TBP (tributyl phosphate) or DBBP (dibutyl
butyl phosphonate) is enhanced by high nitrate salt concentrations in the
aqueous phase, particularly at HNO_3 concentrations below ∿ 1.0 $\underline{N}$.

II. Solvent Extraction Experiments

Solvent extraction studies were done on two feed samples, dissolved
hydroxide cake, SSA, and evaporator supernate, SSB. SSA was prepared by dis-
solution of hydroxide cake by slow addition of concentrated HNO_3, adjust-
ment of the final acidity to ∿ 0.5 $\underline{N}$ by addition of H_2O and/or HNO_3, and
clarification by filtration. To prepare SSB, some supernate was titrated,
acid adjusted to ∿ 0.5 $\underline{N}$ by NaOH and water addition, then clarified by fil-

tration. No appreciable solids were observed on the clarification filters for either solution. Compositions of these feeds are listed in Table III.

After exploratory tests on a solution simulating SSA but with Nd as an Am stand-in, the extraction system 30 v/o DBBP-70 v/o Isopar H (a commercially available odorless kerosene) was chosen for tests on the SSA and SSB feeds.

All experiments were done in separatory funnels by mixing equal volumes of the organic and aqueous phases for ∿ 5 minutes, allowing to settle ∿ 5 minutes, and then separating. Samples of each of the two phases were analyzed for the constituents of interest. All reported results are those of experiments that displayed acceptable mass balances which were used as a check of experiment validity.

III. Results and Discussion

Extraction and stripping experiments were designed to provide data for several a eas of interest.

A. Am distribution coefficients[31] as a function of the equilibrium aqueous phase acidity of the feed were obtained. These results are shown in Figure 11. The K_D's vary from 6 to 60 for acidities from 0.4 to 0.03 $\underline{N}$ respectively. The data points designated, B-X, are for SSB which is thought to have a higher NO_3^- salt concentration than SSA, so these appear above the curve of the SSA points.

B. Am distribution coefficients as a function of the equilibrium aqueous phase acidity of non-salted strip solutions were studied. The results are given in Figure 12. Here, the K_D's vary from 0.25 to 0.01 for acidities from 0.4 to 0.03 $\underline{N}$. These low values from about 0.1 $\underline{N}$ downward are most valuable for stripping.*

C. Acid distribution between the equilibrated organic and feed phases is shown in Figure 13. These data are necessary for maintaining proper acid conditions during the extraction process. Previous workers[30] have shown that HNO_3 forms a 1:1 complex with DBBP, so it is evident that both acid and Am move into the extractant. Although this reduction of feed acidity helps Am extraction by increasing the K_D, excessive feed acidity reduction may cause solids to form in the system by hydrolysis of the metal salts; therefore, it must be kept under control.

* Also shown are data points for acidities of 3.5 and 7 $\underline{N}$.

D. The extractive effectiveness of DBBP-Isopar H was tested under several conditions: as received; pre-acidified only; and washed with Na_2CO_3 solution and then pre-acidified. The results for all three systems were the same within experimental error.

E. Tests of the reuse of the solvent system after the Am was stripped showed no anomalies in the Am K_D.

F. The i·ed solutions for the extractions contained appreciable amounts of Pb, U, and Pu, so analyses were done to determine their course through the extraction-strip cycles. It was found that with either water or 0.01 $\underline{N}$ HNO_3 strips, the Am product solution did not have appreciable Pb contamination; however, the Pu and the U contamination was unacceptably high. It was decided that stripping with higher concentrations of HNO_3 would offer two advantages; first, the extraction coefficient for U and Pu would be larger, and second, the Am complexes with the HNO_3 in the aqueous phase at higher HNO_3 concentrations[26] so should strip well. Tests showed that contamination of the Am by Pu was essentially eliminated by stripping the Am with 3.65 to 7.3 $\underline{N}$ HNO_3 whereas that by U was reduced to acceptably low levels. The Am stripping K_D's for these strips were 0.22.

G. Since the U and Pu are readily extracted from the aqueous feed and are forced to stay in the organic during the Am stripping, the solvent will eventually be loaded with them. Tests of solvent clean-up showed that these could be quickly and cleanly stripped with 5 w/o Na_2CO_3, 9 w/o $NaHCO_3$, or 20 w/o $(NH_4)_2CO_3$ solutions with no solid formation.

IV. Proposed Solvent Extraction Process for Am Recovery

These studies result in the proposed solvent extraction recovery process for Am as given in the flowsheet of Figure 14. Solvent clean-up and recycle are included. The Am product stream is to enter the present AmO_2 production line directly behind the anion exchange Pu removal operation.

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31. Distribution coefficient is defined as the ratio of the Am concentration in the organic phase to its concentration in the aqueous phase when the two are in equilibrium.

T A B L E   1

COMPOSITION OF PEROXIDE FILTRATE BATCHES FROM FFTF

The filtrates were approximately 0.03 $\underline{M}$ in F^- and 0.056 $\underline{M}$ in $SO_4^=$. These filtrates were dripped into approximately 80 moles of NaOH. Values are mg/ℓ unless otherwise noted.

Batch No. Element	1049	1127
Am	4.47 g	7.37 g
Pu	0.49 g	0.54 g
Ag	< 0.15	< 0.1 g
Al	500	200
B	25	4
Ba	0.5	0.8
Be	< 0.05	< 0.04
Bi	< 1.5	< 1
Ca	< 25	< 30
Cd	< 5	< 4
Co	< 1.5	< 1
Cu	5	1.6
Cr	5	4
Ga	< 0.5	< 0.4
Ge	< 0.5	< 0.4
Fe	15	16
In	< 1.5	< 1
K	< 50	< 40
Li	< 5	< 4
Mg	25	8
Mn	1	0.8
Mo	3	< 1
Na	75	20
Ni	10	2
Nb	< 5	< 4
P	< 5	< 4
Pb	2	4
Sb	< 1.5	< 1
Si	20	20
Sn	< 3	< 2
Sr	0.25	0.6
Ta	< 50	< 40
Ti	1.5	< 0.4
Tl	< 1.5	< 1
V	< 1.5	< 1
Zn	< 3	< 2
Zr	< 0.5	< 0.4

T A B L E   2

PRODUCT ANALYSIS OF LASL AmO_2

Results are in ppm except for Am and Pu which are reported in Wt. %.

Batch No. Element	AmO_2 20	AmO_2 24	AmO_2 27
Am	87.3 %	87.5 %	86.0 %
Pu	0.076%	2.2 %	0.32 %
Al	7	22	7
Ca	27	840	?4
Cl	20	35	27
Cr	19	60	25
F	3	5	17
Fe	40	73	53
K	12	24	8
Mg	2	4	17
Mn	2	6	3
Na	50	140	580
Ni	9	13	14
Np	500	450	450
P	3	1	6
Pb	370	34	5
S	--	5	--
Si	94	980	110
Th	--	74	--
U	< 10	365	38
Y	110	28	360

TABLE 3

FEED SOLUTION COMPOSITIONS

SPECIES	(Dissolved Hydroxide Cake) SSA		(Evaporator Supernate) SSB	
	g/l	M	g/l	M
Am	0.46		0.34	
Pu	0.15		0.029	
U	3.9		0.81	
Pb	1.3		0.45	
Th	---		0.004	
Na		1.		3.3
Al		0.4		0.67
Ca		0.3		0.23
Fe		0.21		0.11
Mg		0.15		0.07
Cr		0.06		0.02
Ni		0.06		0.01
K		---		0.05
H^+		0.44		0.5
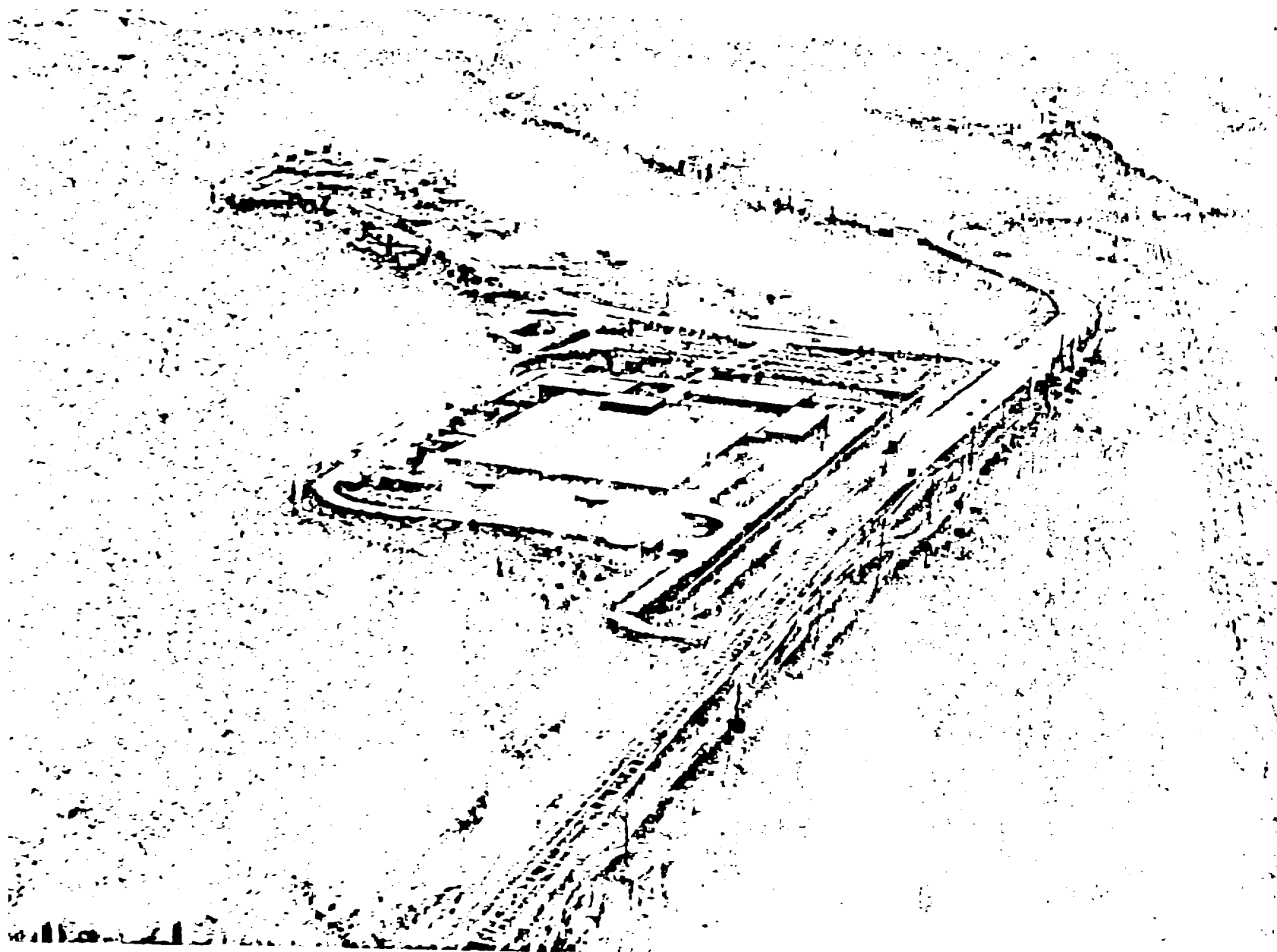
Figure 1. TA-55 Plutonium Facility at Los Alamos Scientific Laboratory.

LASL FFTF PuO₂ PRODUCTION
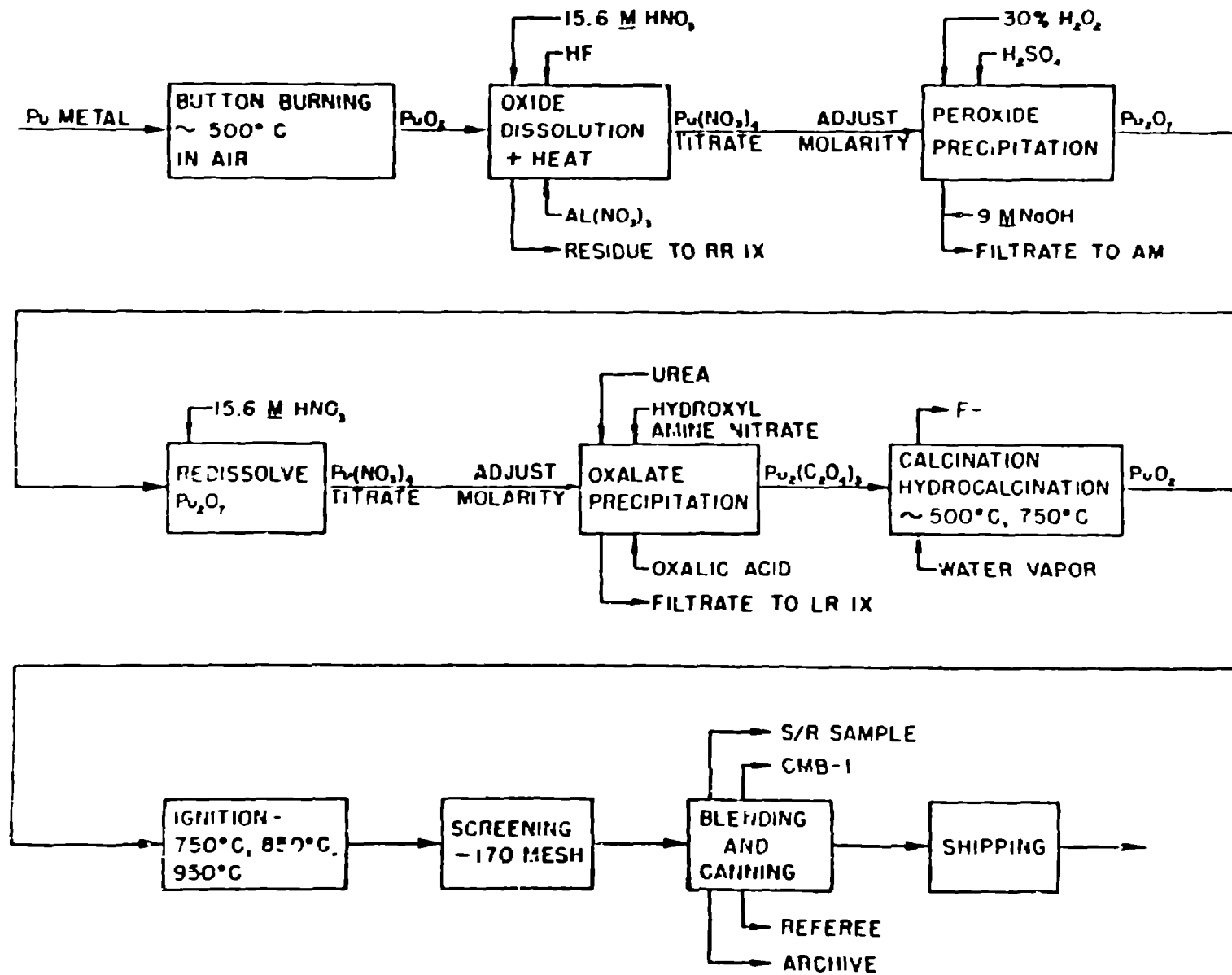
Figure 2. Flowsheet for FFTF oxide production.

LASL AMERICIUM OXIDE PRODUCTION

Hydroxide Slurry from Plutonium Oxide Production → **Filtration & 0.1 M NaOH Wash**

15.6 M Nitric Acid ↓ → **Dissolution**

Filtrate → **Count** → **Discard** / **Recycle**

Acidity Adjustment to 7. M

1. M Oxalic Acid ↓   **Water** ↓

Oxalate Precipitation ← **Distillation & Acidity Adjustment to 0.5 M** ← **Effluent** ← **Anion Exchange Pu Removal**

Resin Clean-up & Recycle Pu to Recovery (IX)

Filtration & 0.1 M Oxalic Acid Wash → **Calcination to Americium Oxide 800° C**

Filtrate to Pu Recovery

Figure 3. Flowsheet for americium oxide production.
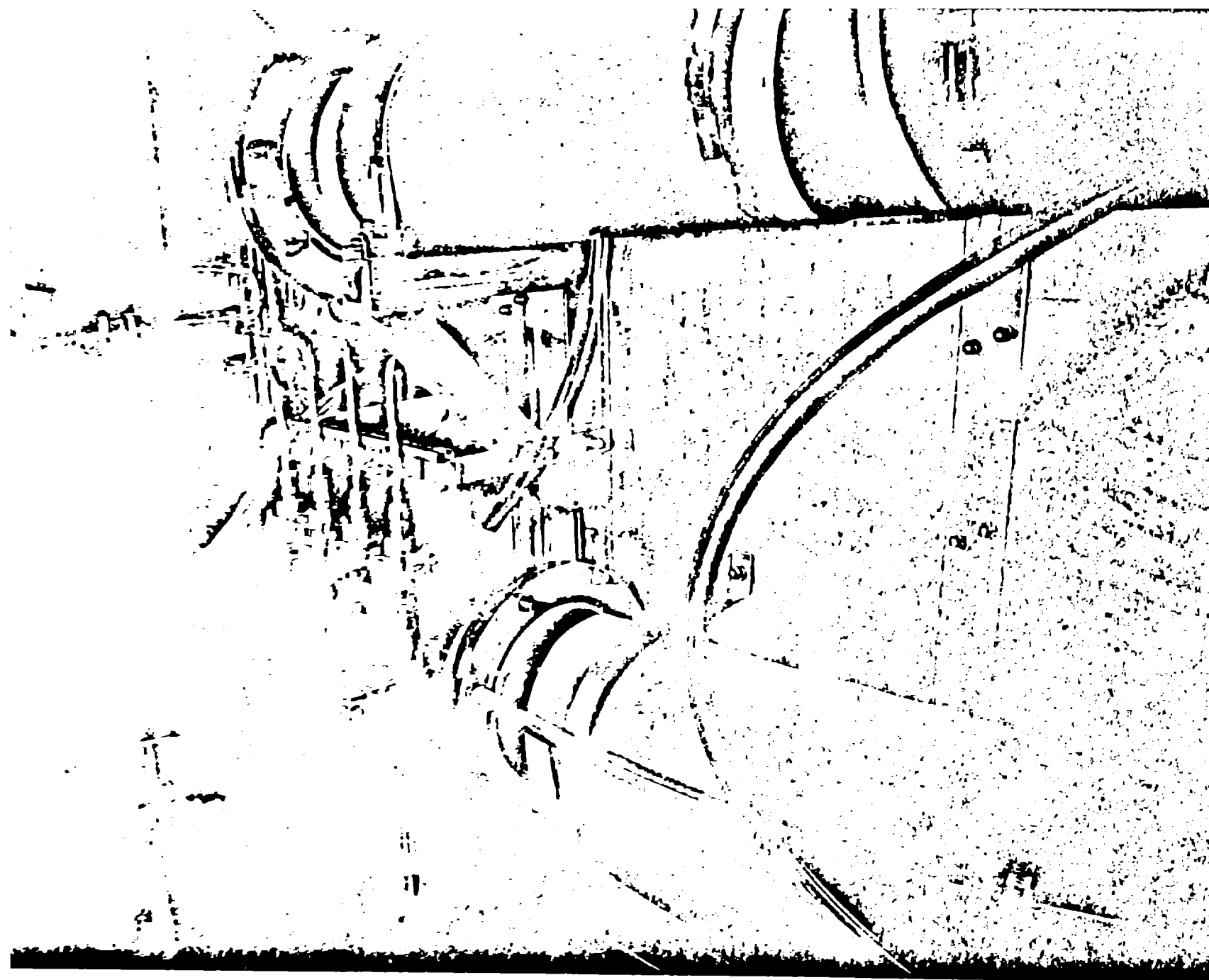
Figure 4. Americium holding tanks (Teflon coated lead shielding).
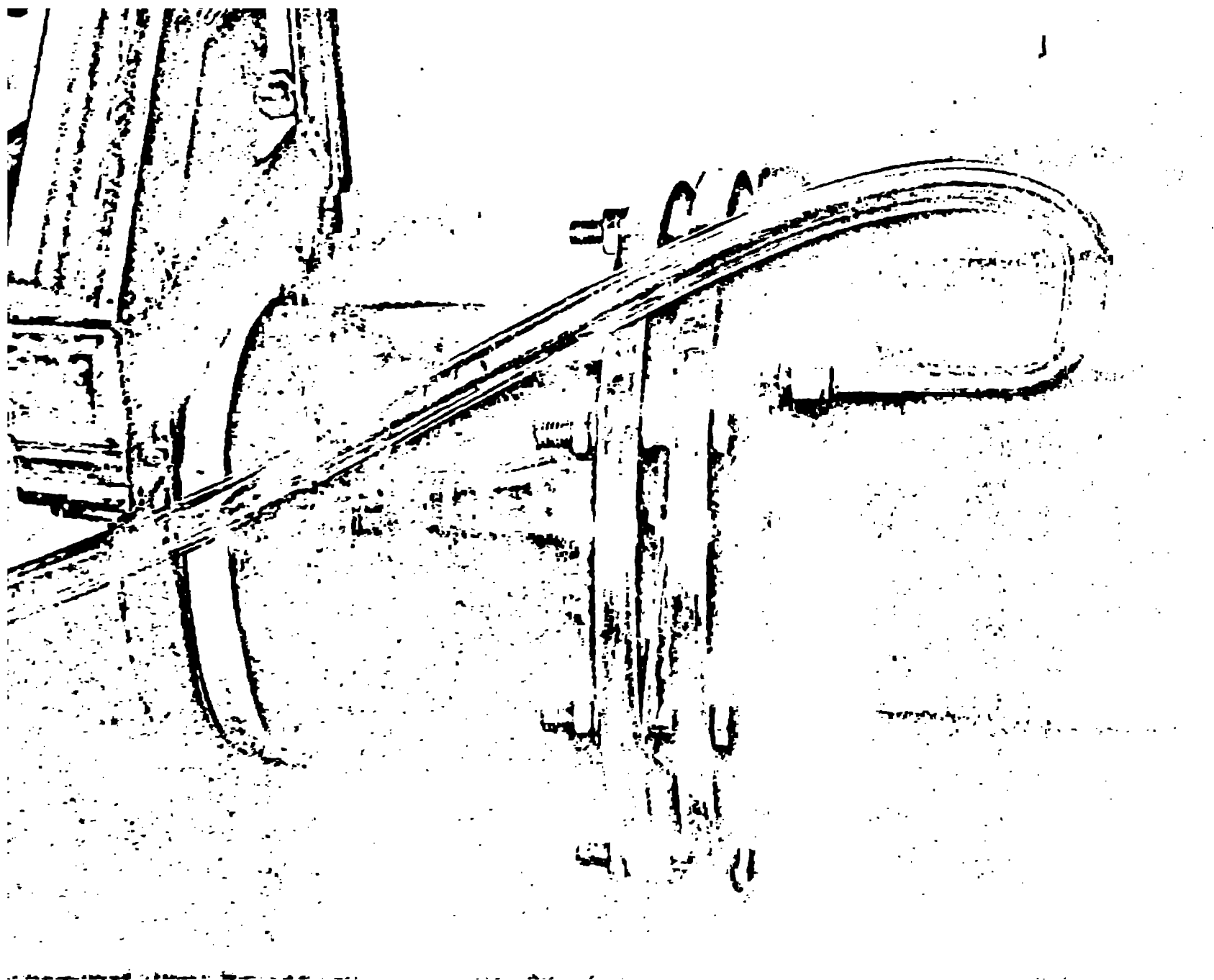
Figure 5. Americium hydroxide slurry after standing 12 hours.
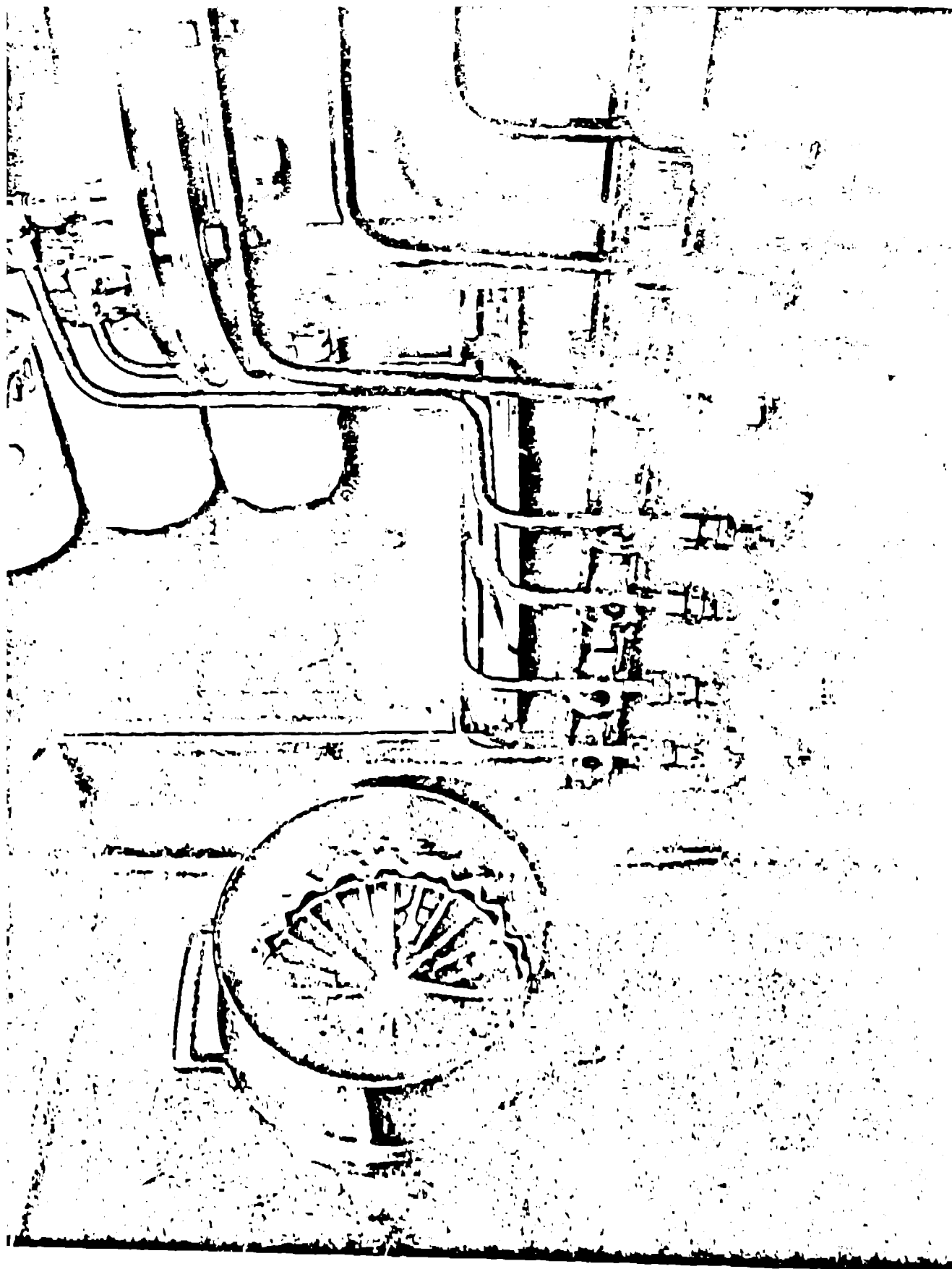
Figure 6. Americium hydroxide in filter boat.
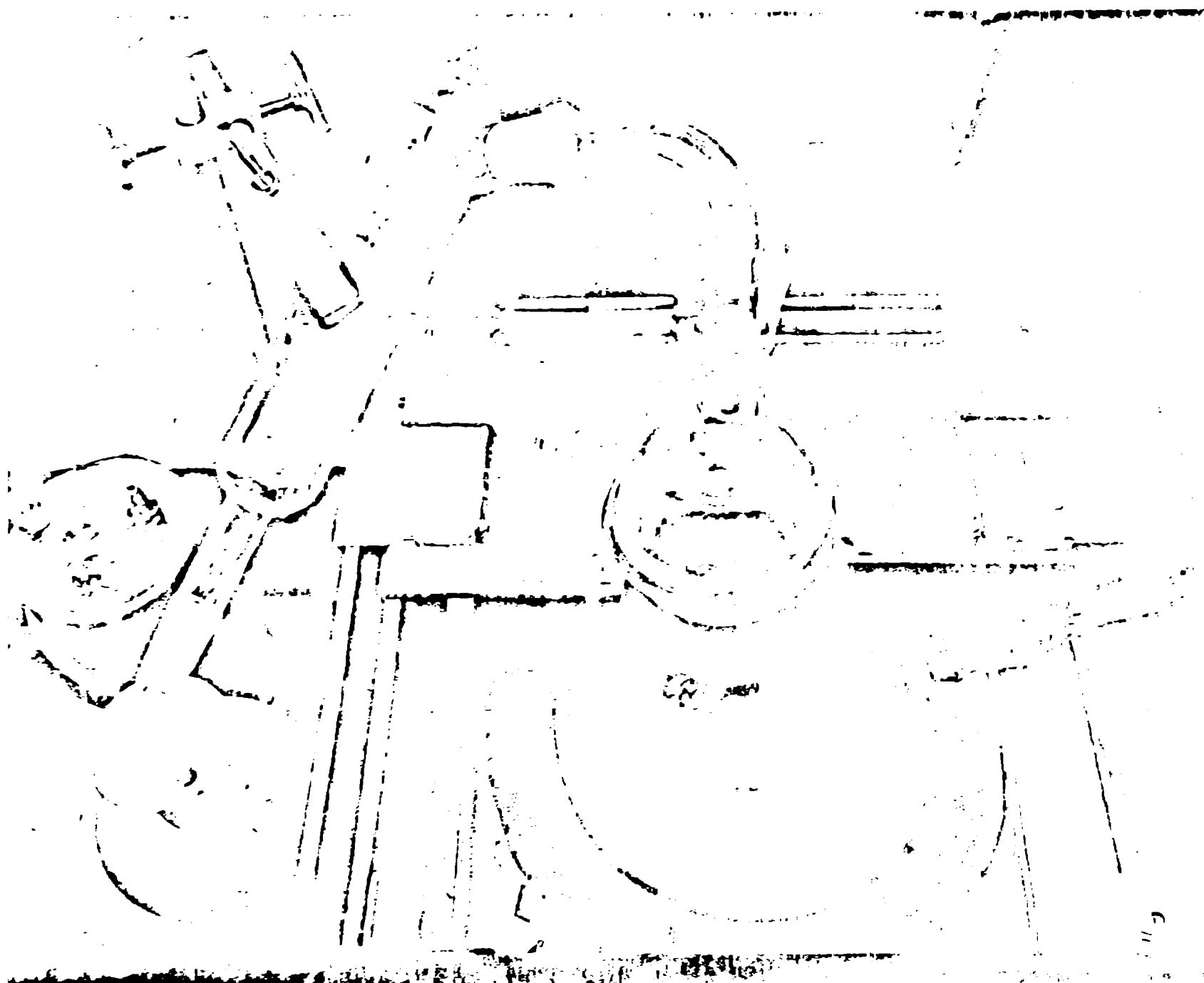
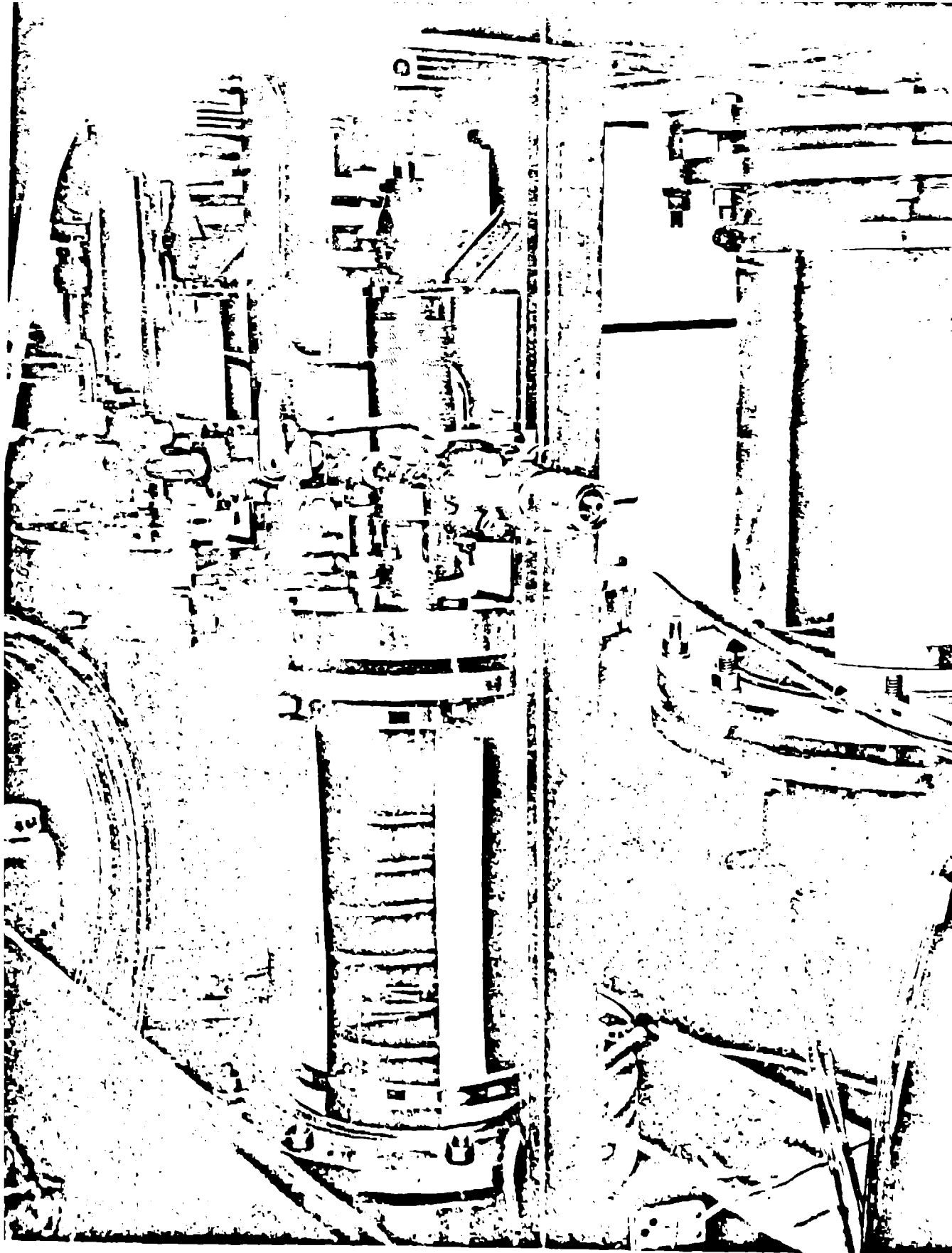Figure 7. Denitrification of americium nitrate solution.
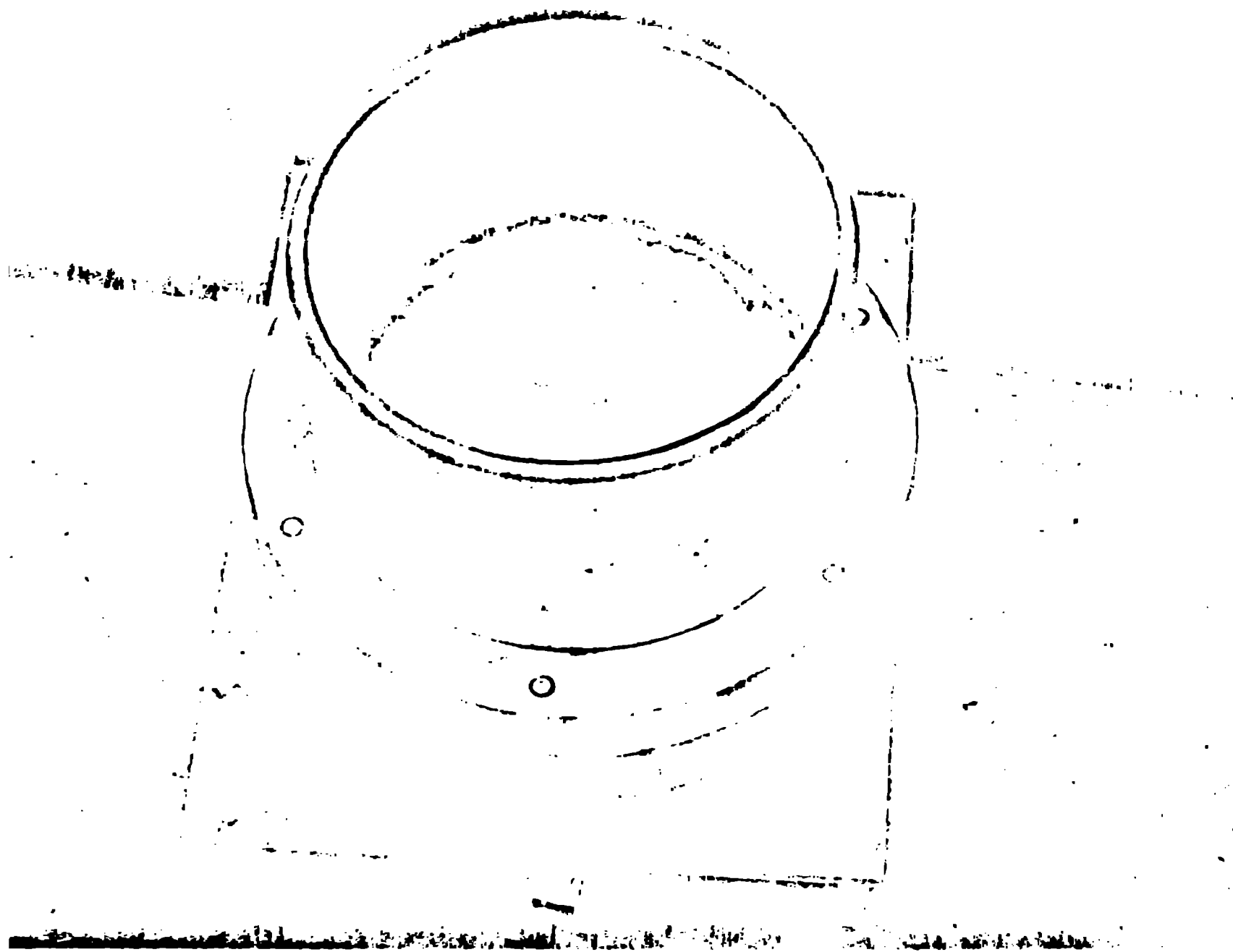
Figure 8. Americium oxalate precipitation vessel.

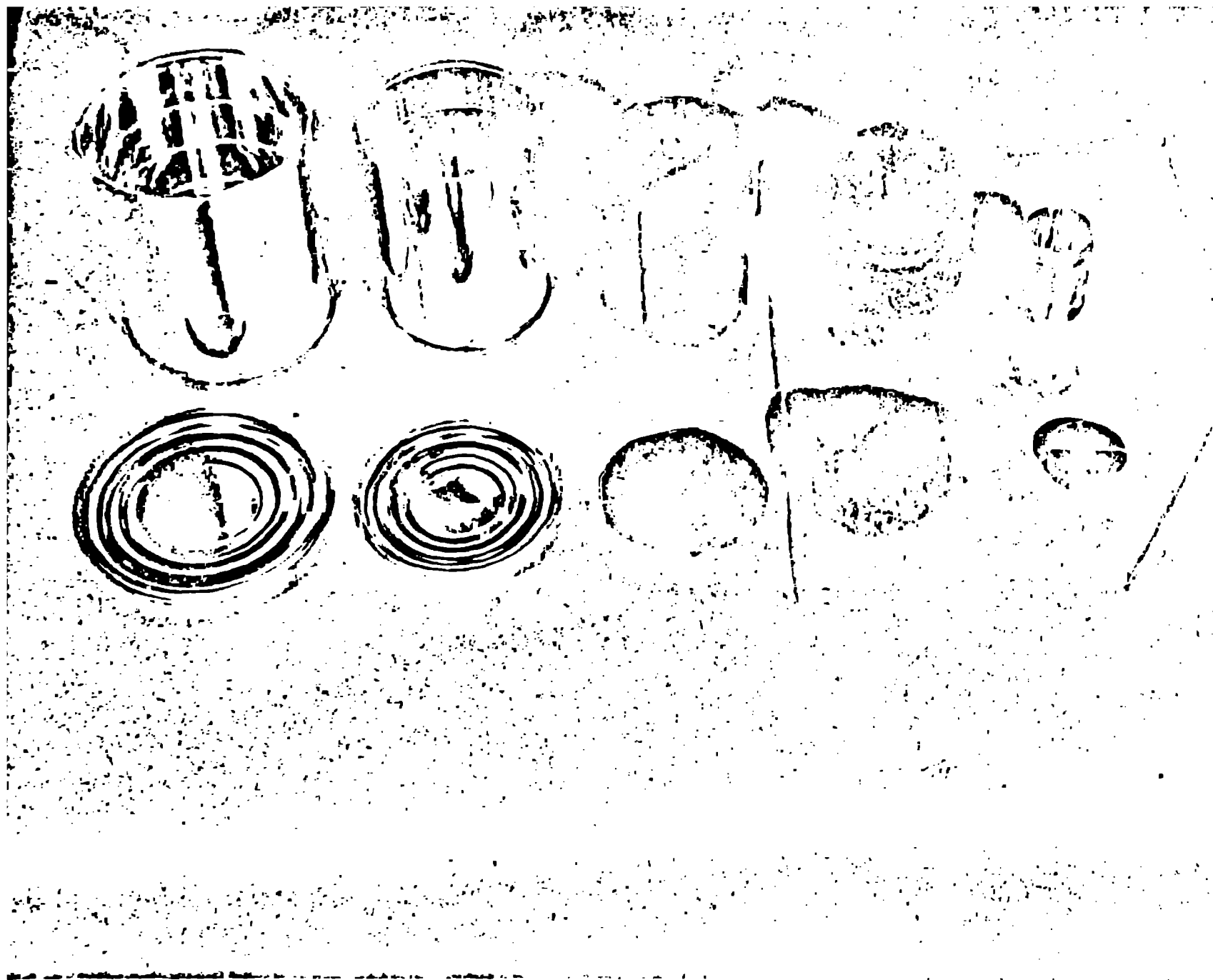Figure 9. Stainless steel filter boat used for americium oxalate.

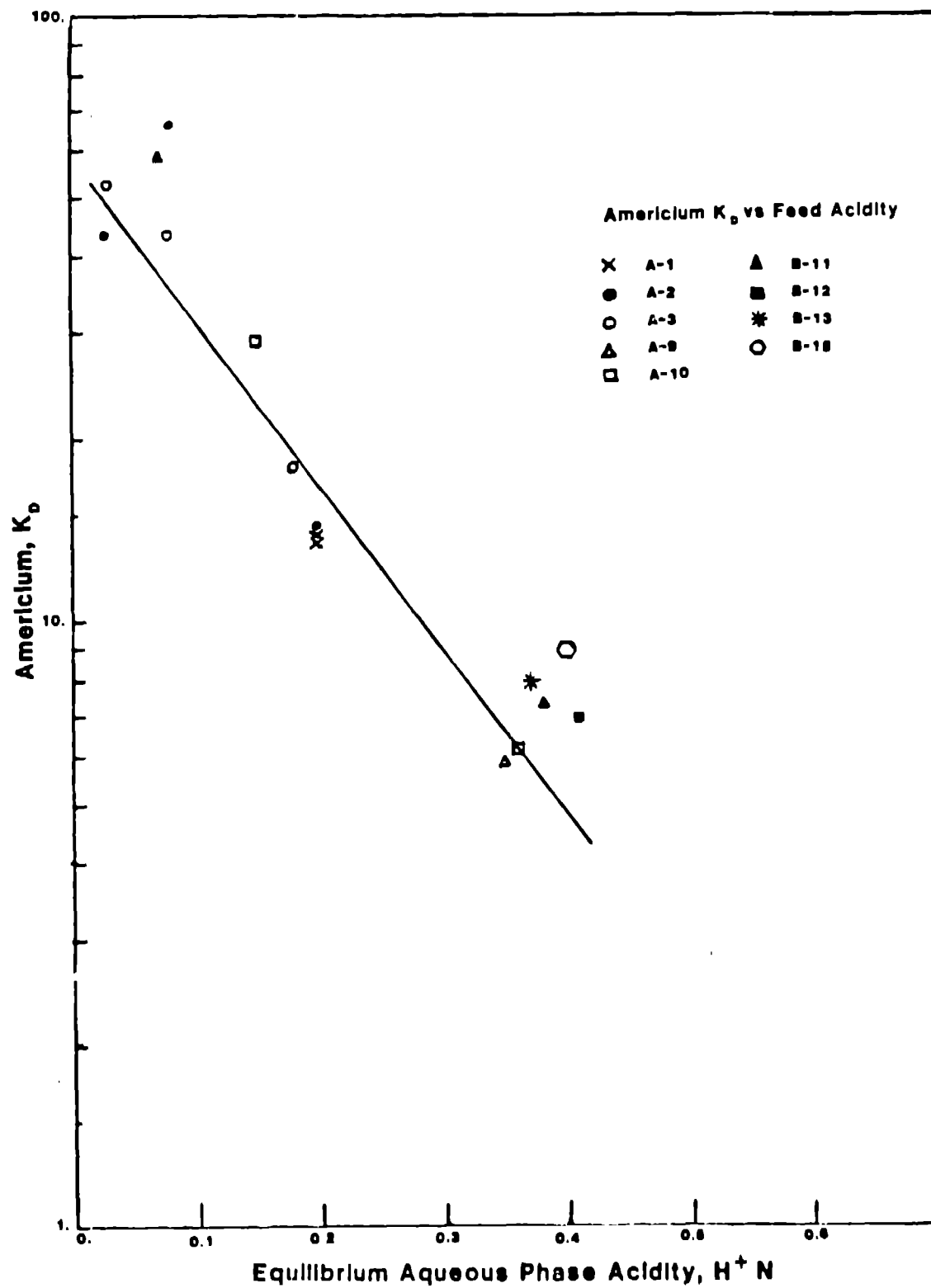Figure 10. Shipping configuration for americium oxide.

Americium K_D vs Feed Acidity

✕	A-1	▲	B-11
●	A-2	■	B-12
○	A-3	✳	B-13
△	A-9	○	B-18
□	A-10		

Americium, K_D
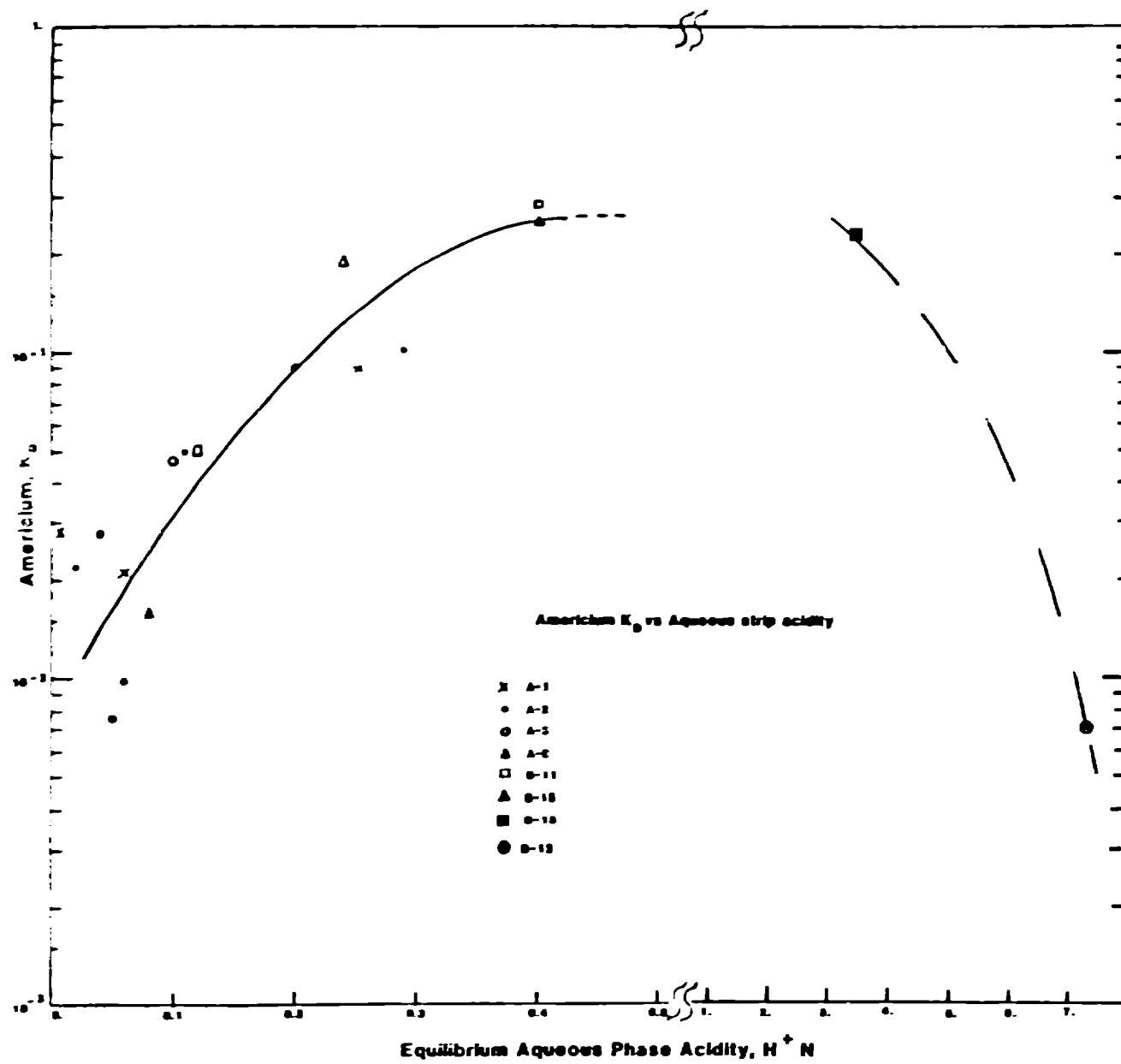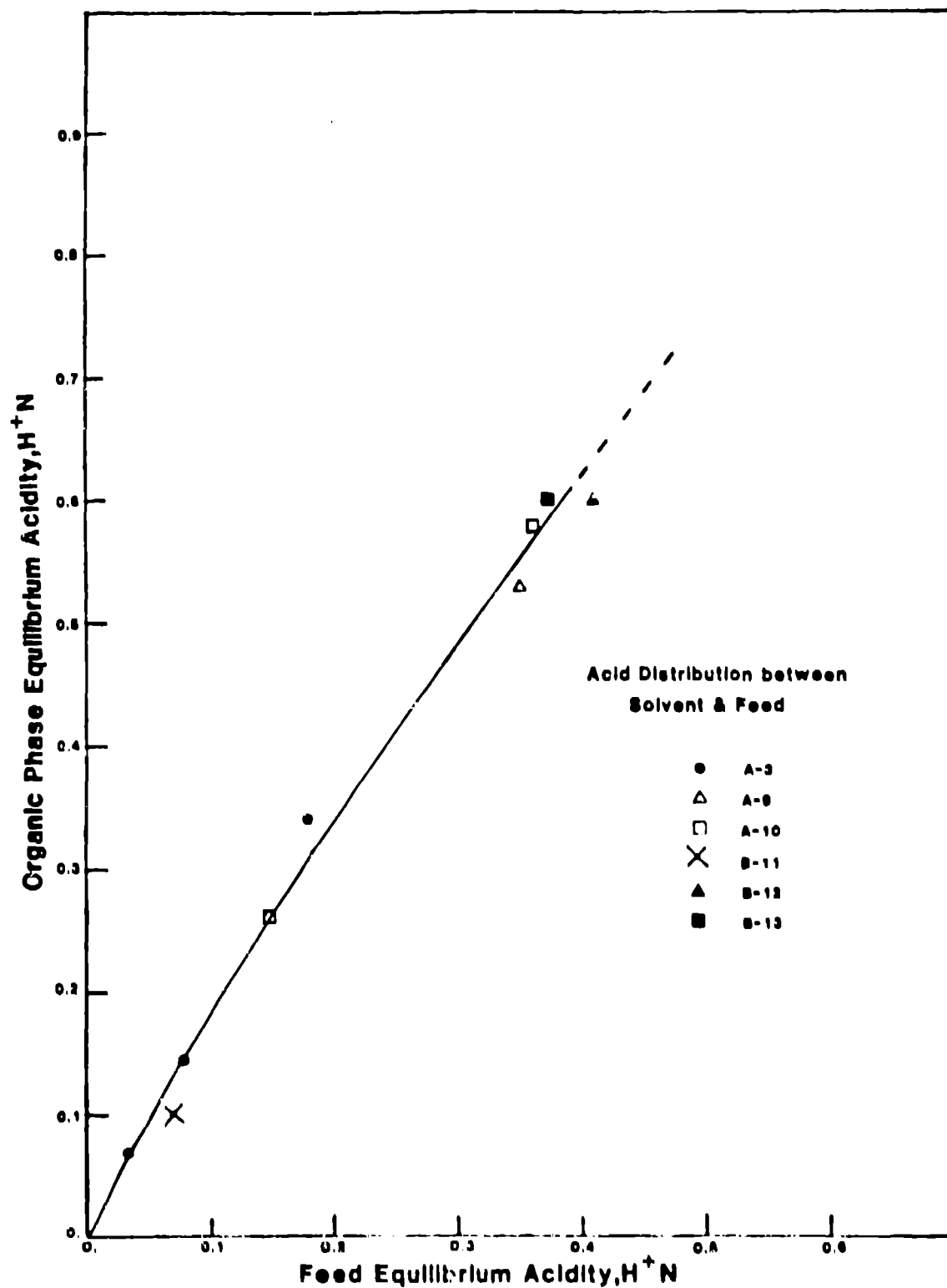
Equilibrium Aqueous Phase Acidity, H^+ N

FIGURE 11

FIGURE 12

FIGURE 11

FIGURE 14

PROPOSED SOLVENT EXTRACTION

PROCESS FOR AMERICIUM